(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
18 March 2004 (18.03.2004)

PCT

(10) International Publication Number
WO 2004/022842 A1

(51) International Patent Classification[7]: D21B 1/02

(21) International Application Number:
PCT/SE2003/001296

(22) International Filing Date: 21 August 2003 (21.08.2003)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
0202629-2 6 September 2002 (06.09.2002) SE

(71) Applicant *(for all designated States except US)*: **STORA ENSO AKTIEBOLAG** [SE/SE]; Åsgatan 22, S-791 80 FALUN (SE).

(72) Inventors; and
(75) Inventors/Applicants *(for US only)*: **PENG, Frank** [SE/SE]; Lagmansvägen 12, S-663 41 HAMMARÖ (SE). **FERRITSIUS, Rita** [SE/SE]; Hemskogsvägen 11, S-791 33 FALUN (SE). **ÄNGSÅS, Ulf** [SE/SE]; Garvaregatan 3E, S-791 70 FALUN (SE).

(74) Agents: **KYLIN, Peter** et al.; Hynell Patenttjänst AB, Patron Carls väg 2, S-683 40 HAGFORS/UDDEHOLM (SE).

(81) Designated States *(national)*: AE, AG, AL, AM, AT (utility model), AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ (utility model), CZ, DE (utility model), DE, DK (utility model), DK, DM, DZ, EC, EE (utility model), EE, ES, FI (utility model), FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK (utility model), SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States *(regional)*: ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:
— *with international search report*
— *with amended claims*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

WO 2004/022842 A1

(54) Title: A METHOD OF PRODUCING MECHANICAL PULP AND THE MECHANICAL PULP THUS PRODUCED

(57) Abstract: A method of producing mechanical pulp, comprising impregnation of fiber material with an enzyme-containing aqueous liquid prior to defibration and refining of the fiber material to produce a mechanical pulp. According to the invention, said enzyme-containing aqueous liquid is a pectinase-containing aqueous liquid. The invention is also related to the mechanical pulp thus produced.

A METHOD OF PRODUCING MECHANICAL PULP AND THE MECHANICAL PULP THUS PRODUCED

TECHNICAL FIELD

The present invention relates to a method of mechanical pulping based on cellulose fiber material after pretreatment of the fiber material with pectinase prior to mechanical defibration. The invention is also related to the mechanical pulp thus produced.

PRIOR ART

In mechanical pulping wood fibers are separated by mechanical defibration, typically in a refiner or a grinder, with or without pretreatment. When pretreatment is applied, the process can be either TMP, where thermal softening is applied; or CTMP, where for instance sulfite is used for the sulfonation of lignin; or APMP, where alkaline peroxide is applied resulting in a combined effect of chemical softening due to alkali and bleaching or brightening due to peroxide. In almost all the mechanical pulping processes, thermal treatment is employed prior to refining to soften the wood material thus leading to reduced energy consumption as well as improved pulp strength. While alkaline treatment is an effective treatment for reducing energy consumption, it also induces darkening reactions that result in poorer optical properties that are essential for paper products based on mechanical pulps. Besides, the use of alkali results in extensive dissolution of wood material, which results in yield loss and necessitates effluent treatment.

In addition to conventional processes, there have been efforts to apply biotechnology to mechanical pulping. In WO 97/40194, a mechanical pulping process using various fungi or enzymes is described. The proposed treatment includes compression of wood chips followed by impregnation with a biological agent prior to refiner treatment. Although a great number of fungi and enzymes are referred to in the patent application, the biological agents used in the given examples are limited either to fungus with ligninase activity, or to enzymes based on xylanase activity aiming for the hemicellulose, and lipase activity specific for the wood resin. The examples show that the enzymatic treatment can reduce the refiner energy by 4 to 10%, in addition to the energy reduction of about 4% due to the compression treatment as compared to the reference. At the same time the strength properties of the pulp are improved when compared at the same pulp freeness.

Another biological pulping process involving pretreatment of wood chips is based on lignin oxidizing fungi treatment (US 5,055,159). The refining energy consumption can be reduced by about 30% at the same freeness. The treatment however requires long incubation time and the pulp produced has a dark color.

Different to the above-mentioned applications, pectinase has been specifically used in post-treatment of mechanical pulps. In US 5,487,812 pectinase is used for alkaline bleached mechanical wood pulp. The purpose of such treatment is however to remove pectin remaining after the pulping process in order to reduce the source of so-called "anionic trash" which is harmful in paper machine operation.

SUMMARY OF THE INVENTION

The present invention aims at providing a method of mechanical pulping requiring significantly reduced refining energy consumption to reach a certain pulp freeness, or pulp strength, as compared to prior art. The invention also aims at improved optical properties in the pulp, such as an improved brightness.

This is accomplished by the method according to the invention, as defined in the claims.

According to the invention, the method comprises pretreatment of the comminuted cellulose fiber material with pectinase, alone or in combination with a chelating agent, for instance DTPA and/or sulfite, followed by mechanical defibration and refining to produce a mechanical pulp.

Pectin is a group of amorphous polysaccharide substances in wood. Although the amount of pectin in softwood and hardwood is normally less than 1%, it is predominantly deposited in the compound middle lamella, and the tori of bordered pit-membranes of coniferous species (Hafren J. and Westermark U.: Nordic Pulp and Paper 16 (4), 284-290, 2001). The main component of pectin is polygalacturonan consisting of galacturonic acid units that to various extend are esterified. When in free acid form, the carboxylic groups of the galacturonan having a negative charge can induce local swelling of the fibers in contact with aqueous solutions. The esterified groups, on the other hand, can be de-esterified through for instance alkaline treatment and thus contributing to swelling.

Given the technical background, the present invention is aimed to utilize the specific features of pectin with respect to its specific location and the potential to induce selective weakening in the fiber wall. Enzyme with specific activity towards pectin is used for the pretreatment of cellulose fiber material. Maceration of the fiber material prior to the enzyme treatment proves an efficient way to get the enzyme in place. To render the fiber separation in the region where chemical weakening occurs, the refining condition is optimized in respect to refining intensity and preheating temperature. The results according to the present invention are a significantly reduced refining energy to reach a given pulp freeness and improved strength and optical properties of the pulp.

According to one aspect of the invention, the pretreatment is preferably assisted by a mechanical compression to facilitate liquor uptake and to give a more even treatment of the material. Compression screw device or twin roll presses can be used at a compression ratio of 1:1 to 8:1. The pretreatment can be further improved if presteaming is conducted prior to the compression.

Impregnation according to the invention follows immediately after the compression and/or thermal pretreatment. The pectinase is charged in an amount required for sufficient treatment, preferably 2,000,000 to 200,000,000 polygalacturonase units/ton material and even more preferred 10,000,000 to 50,000,000 polygalacturonase units/ton. The retention time of treatment is 3 min to 24 hours, preferably 3 to 300 min, even more preferred 15 to 240 min and most preferred 30 to 120 min at a temperature of 20 to 100°C, preferably 35 to 70°C, and more preferably about 50°C. The treatment condition can be adjusted depending on the charge of the pectinase to allow for a sufficient hydrolysis of the pectins. Other pectinase preparations can be used provided that the pectolytic activity is satisfied.

Defibration and refining of the pectinase treated material can be carried out either at conventional TMP conditions and high intensity conditions. A surprising energy reduction effect is obtained. To reach a given pulp freeness of 100 ml CSF, the energy reduction is 400 kWh/t with TMP condition, from 2500 kWh/t without pectinase treatment to 2100 kWh/t with pectinase treatment, or by about 16%. The energy reduction is 150 kWh/t with high intensity condition, from 2150 kWh/t without pectinase treatment to 2000 kWh/t with pectinase treatment, or by about 7%. The combination of high intensity and pectinase treatment gives a total reduction of 500 kWh/t, from 2500 kWh/t to 2000 kWh/t at a pulp freeness of 100 ml CSF, which is 20% in reduction. The shives content decreases more rapidly after the pectinase treatment.

The strength properties remain the same as compared to conventional TMP, and improved by about 10% as compared to high intensity TMP. The brightness of the pulp is also increased by the enzymatic treatment.

While the addition of DTPA or sulfite doesn't contribute to further energy reduction, it increases the brightness by 2 to 5 brightness units. The improved brightness is maintained after peroxide bleaching, together with the benefit of an increased amount of residual peroxide for recycling.

The surprising effects resulting from the pectinase treatment could be explained by a selective weakening of the pectin-enriched region in the fiber wall due to hydrolysis of the pectins, with the consequence of a more efficient fiber separation in defibration and refining of the material. It is evident to those skilled in the art that this process can bring about a significant economical benefit, in terms of improved pulp properties and reduced energy cost. It is also evident to those skilled in the art that any enzyme preparation that contains sufficient pectolytic hydrolysis activity, alone or in combination with other chemicals, can be used for the treatment according to the present invention.

The present invention describes a mechanical pulping process comprising:

DETAILED DESCRIPTION OF THE INVENTION

A) Mechanical pretreatment of the comminuted fiber material with suitable mechanical means, for instance compression with screw device or roll press device, to enable an efficient uptake of aqueous solution into the fiber material during an impregnation immediately following the pretreatment. Replacement of resin-containing liquor in natural cellulose fiber material with added aqueous solution is also positive in a process point of view. The compression ratio is preferably in the range of 1:1 to 8:1, preferably 2:1 to 5:1, where 1:1 means a screw conveyer transporting the fiber material into the impregnation bin. In combination with compression treatment, an initial thermal heating of the fiber material may further facilitate an efficient uptake of the aqueous solution, preferably by use of fresh or recycled steam at atmospheric pressure, for 1 to 30 min, preferably 10 to 20 min. As an alternative, thermal heating followed immediately by impregnation may be used instead of compression treatment when no such facility is available. The pretreatment procedure can also be repeated in one to several stages with the freedom of applying various impregnation chemicals in different stages.

B) Impregnation of the fiber material with pectinase-containing aqueous liquid. Added in connection with or immediately after the pretreatment stage, the enzyme can more easily reach the sites of reaction. In addition to the pectinase enzyme, other chemicals for instance chelating agents such as sulfite, preferably at a charge of 5 to 50 kg/ton, and/or DTPA, preferably at a charge of 1 to 10 kg/ton, may be added in the impregnation to further improve the process. The pectinase-containing liquid may comprise an enzymatic preparation with pectolytic activity for both un-esterified pectins and esterified pectins. Moreover, the aqueous liquid may comprise two or more enzymatic preparations wherein at least one of the preparations has pectinase activity; or the pectinase may be added as a biological agent comprising one or more fungi or bacteria, at least one of which having pectolytic activity. The charge of enzyme is subject to process conditions and cost effectiveness, and lies in the range of 2,000,000 to 200,000,000 polygalacturonase units/ton fiber material, preferably 10,000,000 to 50,000,000 polygalacturonase units/ton. The pH of the impregnation liquor is adjusted by alkali or acid, preferably by caustic soda or mineral acid, to a value optimal for enzymatic reactions, suitably in the pH range of 3 to 10, preferably 4 to 7 and more preferably about 5. The retention time and temperature are also adjusted depending on the process setup and reaction requirements, preferably being 3 to 300 min at a temperature of 20 to 100°C. It is evident to those skilled in the art that any enzyme that contains sufficient pectolytic activity, alone or in combination with other chemicals, can be used in the treatment as described.

C) Defibration and refining of the impregnated fiber material is carried out under optimized conditions. The fiber material is preferably preheated prior to feeding into the pressurized primary refiner, to allow for thermal softening of the fiber wall. The pulping conditions can comprise a refiner rotation speed of 1000 to 3000 rpm, preferably 1500 to 2600 rpm, using either conventional TMP or high refining intensity. Conventional TMP conditions may comprise preheating at 0 and up to 4 or 5 bar with a retention time of 2-10 min in the preheater and a refiner rotation speed of 1200 to 1800 rpm. The pressure 0 bar means atmospheric refining. High intensity conditions may comprise preheating at above 4 or 5 bar and up to 8 bar with a retention time of 3 to 30 sec and a refiner rotation speed above 2000 rpm but usually not above 3000 rpm. The retention time should be matched against the preheating temperature (steam pressure) as high preheating temperature requires shorter retention time. After the primary refining, a secondary refining stage can be used to reach the required pulp freeness. The secondary refining stage may have the same conditions as the primary stage.

Although disc refiners are the defibration and refining equipment used in this study, other mechanical devices can be used for the purpose of defibration and refining. Some of the examples are conical refiners and stone ground wood grinders. The refiners can be of the single disc or double disc type. The refining may be made in a single stage or in multistage. The defibration and refining can also be done at atmospheric pressure, in which case refiner mechanical pulping, or RMP is a more appropriate description of the process instead of TMP.

Although only softwood fiber material is used in this study, any fiber material containing pectins could be treated with this method. The fiber material could be hardwood fiber material, non-wood fibers such as bagasse, bamboo, reed and straw.

The embodiment as described here can be modified according to the source of fiber material and the process set-up.

The pulp obtained can depending on the application of the pulp be subject to further treatment such as washing, screening, post-refining and bleaching according to conventional processes.

BRIEF DESCRIPTION OF THE DRAWINGS

In the following, there will be given a few examples of preferred embodiments according to the invention. The invention is however not limited to these examples. Reference is made to the enclosed drawing diagrams, of which:

Fig. 1 is showing freeness vs. specific refining energy for Example 1,
Fig. 2 is showing freeness vs. specific refining energy for Example 2,
Fig. 3 is showing freeness vs. specific refining energy for Example 3.

EXAMPLES

Mechanical pulps are produced by different chemical treatments, in a pilot refiner plant. The process conditions are as follows.

<u>Material</u>

Wood chips consisting of approximately one third white spruce, one third red and black spruce and one third balsam fir, all of Canadian origin in the Nova Scotia region. The chips are prepared with mill-size chipper and well mixed prior to the trials.

Pretreatment

The chips are pre-steamed at atmospheric pressure for 15 min. After pre-steaming the chips are fed into a plug-screw device with a compression ratio of 4:1. At the outlet of the device, the chips are released into a solution containing various chemicals, including pectinase in the trials according to the invention. The pH of the solution is kept at about 5 adjusted by use of sodium hydroxide or sulfuric acid.

Impregnation and retention

After passing through the impregnation bin, the chips are stored in well-isolated barrels. The retention time is 120 min at a temperature around 50°C. The pectinase used in this study is a highly pectolytic enzyme preparation. This enzyme also hydrolyzes the methyl-esterified galacturonic acid in the pectins. The pectinase used is generated by submerged fermentation of a group of microorganisms containing *Aspergillus aculeatus* and *Aspergillus oryzae*. The charge of pectinase in this study is 30,800,000 polygalacturonase units/ton. The charges of DTPA and sodium sulfite are 4 kg/t and 12 kg/t, respectively, were applicable.

Defibration and refining

The primary refiner is of the Sprout-Bauer 36-1 CP single disc refiner type. Two pulping conditions, conventional TMP and high intensity HI-TMP, are applied. The preheating steam pressure is 2.8 bar for TMP and 5.9 bar for high intensity (HI-TMP). The retention time at preheating is 3.4 min for TMP and 12 sec for HI-TMP. The refiner rotation speed is 1800 rpm for TMP and 2600 rpm for HI-TMP. The secondary refining is carried out with a double disc atmospheric refiner to reach the final freeness levels.

The obtained pulps are evaluated both for strength properties and optical properties including a bleachability study with hydrogen peroxide bleaching.

EXAMPLE 1

In this trial TMP conditions are applied for both samples with the difference that reference, Sample 1, is impregnated only with water adjusted to pH 5.

Sample 1: TMP condition with only water impregnation with pH adjusted to 5.
Sample 2: TMP condition with 30,800,000 polygalacturonase units/t pectinase, pH 5.

It is shown in Table 1 that the specific energy consumption required to reach a given freeness is significantly reduced by the enzymatic treatment. The degree of reduction is

approximately 400 kWh/t at a given freeness, which also can be seen in Figure 1. The pulp properties for those samples are comparable to each other over the freeness range applicable for paper manufacture. The shives content is more rapidly reduced by the pectinase treatment. At a freeness of 57 ml CSF, the shives content is 0.12% as compared to the reference, 0.34%.

Table 1.

	Sample 1					Sample 2			
Freeness, ml CSF	665	230	148	129	57	656	115	57	36
SEC (kWh/t)	902	1889	2028	2237	3078	824	1993	2386	3000
Density (kg/m3)		309,6	344,83	371,75	440,53	205,76	384,62	446,43	462,96
Bulk (cm3/g)		3,23	2,9	2,69	2,27	4,86	2,6	2,24	2,16
Burst Ind. (kPa.m2/g)		1,65	1,98	2,29	3,04	0,34	2,46	3,1	3,35
Tear Ind. (mNm2/g)		10,9	9,6	9,5	7,9	4,8	8,7	8	7,4
Tens Ind. (N.m/g)		33,9	39,9	42,7	53,5	10,1	42,9	55,5	57,4
%Stretch		2,03	2	2,08	2,57	1,1	2,2	2,5	2,67
T.E.A.(J/m2)		28,48	32,7	36,95	57,6	4,6	39,7	28,6	64,3
%Opacity		97	97	97,4	98,4	88,2	96,1	97,5	97,5
Light scatt. coe.(m2/kg)		50,1	52,1	52,8	61,4	35,3	53,9	59,2	65,1
ISO%	44,5	48,8	48,6	49,8	52,3	46,6	50	52,6	52,2
%shives(Pulmac-0.1mm)	15	1,3	0,98	0,8	0,34	13,9	0,6	0,12	0,05

EXAMPLE 2

In this trial the high intensity condition is applied for both samples with the difference that the reference, Sample 3, is impregnated only with water adjusted to pH 5.

Sample 3: HI-TMP condition with only water in the impregnation adjusted to pH 5.

Sample 4: HI-TMP condition with 30,800,000 polygalacturonase units/t pectinase, pH 5.

Table 2.

	Sample 3					Sample 4				
Freeness, ml CSF	714	293	197	126	54	725	254	238	121	72
SEC (kWh/t)	531	1482	1652	1897	2655	412	1362	1462	1793	2344
Density (kg/m3)		311,53	362,32	389,11	454,55		317,46	321,54	387,6	423,73
Bulk (cm3/g)		3,21	2,76	2,57	2,2		3,15	3,11	2,58	2,36
Burst Ind. (kPa.m2/g)		1,47	1,78	2,07	2,96		1,82	1,85	2,6	3,2
Tear Ind. (mNm2/g)		10,1	8,3	8,5	7,7		11,1	11,1	9,4	9,2
Tens Ind. (N.m/g)		30,2	34,9	40,8	51,6		34,7	35,4	47,3	53,3
%Stretch		1,97	2,08	2,03	2,63		2	2,1	2,5	2,54
T.E.A.(J/m2)		24,73	29,74	35,27	58,53		30,1	32,4	49,3	57,3
%Opacity		94,6	95,4	96,7	97,2		95,3	94,8	96,6	97,1
Light scatt.coe.(m2/kg)		41,2	49,3	61,6	62,6		49,4	48,9	59,3	60,8
ISO%	45,3	50,3	50,1	52	53,9	44,8	50,6	50,8	51,6	54,4
%shives(Pulmac-0.1mm)	8,42	0,62	0,5	0,38	0,04	8,76	0,52	0,4	0,14	0,06

It is shown in Table 2 and Figure 2 that the enzymatic treatment has a surprising effect on energy reduction also with the high intensity condition. To reach a given freeness of down to about 100 ml CSF, the specific energy consumption is reduced by about 150 kWh/t. The reduction is less pronounced at lower freeness. The pulp properties at the same time are improved by about 10% both for tensile strength and tear strength, while the optical properties remain at the same level. The potential of the enzymatic treatment makes is obvious to people skilled in the art that even better strength can be achieved if the same amount of energy is applied to the enzymatically treated pulps, or even less energy is required if the same strength properties are to be acquired.

EXAMPLE 3

In this trial the HI-TMP condition is applied for all the samples with Sample 3 and Sample 4 from Example 2 as references.

Sample 3: HI-TMP condition with only water in the impregnation adjusted to pH 5.

Sample 4: HI-TMP condition with 30,800,000 polygalacturonase units/t pectinase, pH 5.

Sample 5: HI-TMP condition with 30,800,000 polygalacturonase units/t pectinase, 4 kg/t DTPA, pH 5.

Sample 6: HI-TMP condition with 30,800,000 polygalacturonase units/t pectinase, 4 kg/t DTPA and 12 kg/t sodium sulfite, pH 5.

Table 3

	Sample 3				Sample 4				Sample 5				Sample 6			
Freeness, ml CSF	293	197	126	54	254	236	121	72	201	148	87	52	184	150	97	55
SEC (kWh/t)	1482	1652	1897	2655	1362	1462	1793	2344	1539	1681	2022	2676	1934	2082	2303	3040
Density (kg/m3)	311,53	362,32	389,11	454,55	317,46	321,54	387,6	423,73	340,14	362,32	413,22	446,43	384,62	358,42	398,41	436,68
Bulk (cm3/g)	3,21	2,76	2,57	2,2	3,15	3,11	2,58	2,36	2,94	2,76	2,42	2,24	2,6	2,79	2,51	2,29
Burst ind. (kPa.m2/g)	1,47	1,78	2,07	2,86	1,82	1,85	2,6	3,2	1,87	2	2,63	3,1	2,3	2,24	2,61	3
Tear ind. (mNm2/g)	10,1	8,3	8,5	7,7	11,1	11,1	9,4	9,2	8,6	8,8	8,5	7,8	9,8	9,5	10,3	7,4
Tens ind. (N.m/g)	30,2	34,9	40,8	51,6	34,7	35,4	47,3	53,3	37,1	41,3	47,1	52,8	41,7	41,3	47,1	51,1
%Stretch	1,97	2,08	2,03	2,63	2	2,1	2,5	2,54	2,2	2,1	2,56	2,7	2,1	2,5	2,6	2,9
T.E.A.(J/m2)	24,73	29,74	35,27	58,53	30,1	32,4	49,3	57,3	33	37,9	48,3	57	35,2	40,1	47,2	56,6
%Opacity	94,6	95,4	96,7	97,2	95,3	94,8	96,6	97,1	94,1	95,4	98	98,6	93,2	94,2	95,2	95,5
Light scatt.coe.(m2/kg)	41,2	49,3	61,6	62,6	49,4	48,9	59,3	60,8	52,5	55,6	58,2	60,2	49,3	53,6	57,8	60,1
ISO%	50,3	50,1	52	53,9	50,6	50,8	51,6	54,4	52,1	52,9	54	56	55,4	55,8	56,3	58,8
%shives(Pulmac-0.1mm)	0,62	0,5	0,38	0,04	0,52	0,4	0,14	0,06	0,68	0,54	0,3	0,07	1	0,68	0,48	0,09

The refining energy consumption, SEC, is not affected by additional charge of DTPA or DTPA/sulfite. The SEC is at the same level for all samples with pectinase treatment, about 150 kWh/t lower compared to Sample 3, i.e. high intensity condition without pectinase treatment, also see Figure 3. A surprising effect is however obtained in the brightness of unbleached pulp. With DTPA together with pectinase, the brightness is increased by about 2 units ISO. With a combined of pectinase, DTPA and sulfite, the brightness improvement is even higher, 4-5 units ISO. The brightness improvement is fairly constant in the entire freeness range.

EXAMPLE 4

Pulps with different treatments are bleached with alkaline peroxide with following bleach conditions:

- Pretreatment with 7 kg DTPA/t at 7% consistency, 90°C, pH 5 for 45 min before dewatering to 30%.
- Bleaching at 25% consistency at 30kg/t hydrogen peroxide charge, 80°C for 90 min. Three sodium hydroxide charges, 10, 20 and 30kg/t, are used to find the optimal brightness. The optimal brightness and corresponding residual peroxide are given as bleach results.

Sample 7: TMP condition with only water impregnation with pH adjusted to 5.
Sample 8: TMP condition with 30,800,000 polygalacturonase units/t pectinase, pH 5.
Sample 9: HI-TMP condition with only water in the impregnation adjusted to pH 5.
Sample 10: HI-TMP condition with 30,800,000 polygalacturonase units/t pectinase, pH 5.
Sample 11: HI-TMP condition with 30,800,000 polygalacturonase units/t pectinase, 4 kg/t DTPA, pH 5.
Sample 12: HI-TMP condition with 30,800,000 polygalacturonase units/t pectinase, 4 kg/t DTPA and 12 kg/t sodium sulfite, pH 5.

Table 4

	Unbl. ISO%	Bleached ISO%	Res. H2O2 %
Sample 7	52,3	68,1	7,5
Sample 8	54,7	70,6	16,8
Sample 9	53,9	68,3	5,6
Sample 10	54,4	71,9	19,9
Sample 11	56.0	71,4	12,9
Sample 12	59,5	74,3	25,3

It is shown in Table 4 that the brightness improvement is maintained after the enzymatic treatment. The improvement is about 2.5 brightness units for the TMP condition, and 3.6 units for the HI-TMP condition. Combination of DTPA and sulfite in the impregnation gives further improvement to a brightness of 74.3%ISO, compared to the TMP reference of 68.1%ISO.

Noticeably, the residual peroxide is much higher with the enzymatically treated samples. This is beneficial because the residual chemicals are recycled back to the process after bleaching. The need of make-up peroxide is reduced accordingly.

The invention is not limited by the embodiments described above, but may be varied within the scope of the claims.

CLAIMS

1. A method of producing mechanical pulp, comprising impregnation of fiber material with an enzyme-containing aqueous liquid prior to defibration and refining of the fiber material to produce a mechanical pulp, characterised in that said enzyme-containing aqueous liquid is a pectinase-containing aqueous liquid.

2. A method according to claim 1, characterised in initial compression and/or thermal pretreatment of the fiber material, preferably by mechanical compression and/or steaming, before the impregnation.

3. A method according to claim 2, characterised in that steaming is carried out, preferably at atmospheric pressure, for 1 to 30 min, preferably 10 to 20 min.

4. A method according to claim 2 or 3, characterised in that compression is performed by a compression screw or a twin roll press, with a compression ratio of 1:1 to 8:1, preferably 2:1 to 5:1.

5. A method according to anyone of the preceding claims, characterised in that the pectinase-containing liquid comprises an enzymatic preparation with pectolytic activity for both pectins and esterified pectins.

6. A method according to anyone of the preceding claims, characterised in that the aqueous liquid comprises two or more enzymatic preparations wherein at least one of the preparations has pectinase activity.

7. A method according to anyone of claims 1-5, characterised in that the pectinase is added as a biological agent comprising one or more fungi or bacteria, at least one of which having pectolytic activity.

8. A method according to anyone of the preceding claims, characterised in that the pectinase arises from a group of microorganisms containing *Aspergillus aculeatus* and *Aspergillus oryzae*.

9. A method according to anyone of the preceding claims, characterised in that the charge of pectinase is 2,000,000 to 200,000,000 polygalacturonase units/ton fiber material, preferably 10,000,000 to 50,000,000 polygalacturonase units/ton.

10. A method according to anyone of the preceding claims, characterised in that the aqueous liquid comprises at least one chelating agent, preferably diethylenetetraminepentaacetic acid at a charge of 1 to 10 kg/ton and/or sulfite at a charge of 5 to 50 kg/ton.

11. A method according to anyone of the preceding claims, characterised in that a retention time after uptake of the impregnation liquid is 3 min to 24 hours, preferably 15 to 240 min, and more preferably 30 to 120 min.

12. A method according to claim 11, characterised in that a temperature in the retention after uptake of the impregnation liquid is 20 to 100°C, preferably 35 to 70°C, and more preferably about 50°C.

13. A method according to anyone of the preceding claims, characterised in that a pH in the impregnation liquid is 3 to 10, preferably 4 to 7, and more preferably about 5.

14. A method according to anyone of the preceding claims, characterised in that the defibration and refining of the fiber material is performed by use of single disc, double disc or conical refiners in one or multi stages.

15. A method according to claim 14, characterised in that a refiner rotation speed is 1000 to 3000 rpm, preferably 1500 to 2600 rpm.

16. A method according to claim 14, characterised in that the fiber material is preheated for 2 to 10 min before refining, that a refiner pressure is from atmospheric up to 5 bar, preferably up to 4 bar and that a refiner rotation speed preferably is 1200 to 1800 rpm.

17. A method according to claim 14, characterised in that the fiber material is preheated for 3 to 30 sec before refining, that a refiner pressure is from 4 to 8 bar, preferably 5 to 8 bar and that a refiner rotation speed preferably is above 2000 rpm.

18. A method according to anyone of the preceding claims, characterised in that said fiber material is softwood chips or hardwood chips.

19. A method according to anyone of claims 1-17, characterised in that the fiber material is non-wood fiber material including bagasse, bamboo, reed and straw.

20. A method according to anyone of the preceding claims, characterised in that the pulp obtained after defibration and refining is bleached, preferably with alkaline peroxide, to obtain bleached pulp having high brightness.

21. Mechanical pulp, characterised in that it has been produced according to any one of claims 1-20.

AMENDED CLAIMS

[received by the International Bureau on 20 January 2004 (20.01.04);
original claims 1-2 amended; claims 3-21 unchanged (3 pages)]

CLAIMS

A method of producing mechanical pulp, comprising impregnation of fiber material with an enzyme-containing aqueous liquid prior to defibration and refining of the fiber material to produce a mechanical pulp, said enzyme-containing aqueous liquid being a pectinase-containing aqueous liquid, characterised in initial compression of the fiber material.

2. A method according to claim 1, characterised in that said initial compression of the fiber material is a mechanical compression, preferably combined with a thermal pretreatment of the fiber material, preferably by steaming, before the impregnation.

3. A method according to claim 2, characterised in that steaming is carried out, preferably at atmospheric pressure, for 1 to 30 min, preferably 10 to 20 min.

4. A method according to claim 2 or 3, characterised in that compression is performed by a compression screw or a twin roll press, with a compression ratio of 1:1 to 8:1, preferably 2:1 to 5:1.

5. A method according to anyone of the preceding claims, characterised in that the pectinase-containing liquid comprises an enzymatic preparation with pectolytic activity for both pectins and esterified pectins.

6. A method according to anyone of the preceding claims, characterised in that the aqueous liquid comprises two or more enzymatic preparations wherein at least one of the preparations has pectinase activity.

7. A method according to anyone of claims 1-5, characterised in that the pectinase is added as a biological agent comprising one or more fungi or bacteria, at least one of which having pectolytic activity.

8. A method according to anyone of the preceding claims, characterised in that the pectinase arises from a group of microorganisms containing *Aspergillus aculeatus* and *Aspergillus oryzae*.

9. A method according to anyone of the preceding claims, characterised in that the charge of pectinase is 2,000,000 to 200,000,000 polygalacturonase units/ton

fiber material, preferably 10,000,000 to 50,000,000 polygalacturonase units/ton.

10. A method according to anyone of the preceding claims, characterised in that the aqueous liquid comprises at least one chelating agent, preferably diethylenetetraminepentaacetic acid at a charge of 1 to 10 kg/ton and/or sulfite at a charge of 5 to 50 kg/ton.

11. A method according to anyone of the preceding claims, characterised in that a retention time after uptake of the impregnation liquid is 3 min to 24 hours, preferably 15 to 240 min, and more preferably 30 to 120 min.

12. A method according to claim 11, characterised in that a temperature in the retention after uptake of the impregnation liquid is 20 to 100°C, preferably 35 to 70°C, and more preferably about 50°C.

13. A method according to anyone of the preceding claims, characterised in that a pH in the impregnation liquid is 3 to 10, preferably 4 to 7, and more preferably about 5.

14. A method according to anyone of the preceding claims, characterised in that the defibration and refining of the fiber material is performed by use of single disc, double disc or conical refiners in one or multi stages.

15. A method according to claim 14, characterised in that a refiner rotation speed is 1000 to 3000 rpm, preferably 1500 to 2600 rpm.

16. A method according to claim 14, characterised in that the fiber material is preheated for 2 to 10 min before refining, that a refiner pressure is from atmospheric up to 5 bar, preferably up to 4 bar and that a refiner rotation speed preferably is 1200 to 1800 rpm.

17. A method according to claim 14, characterised in that the fiber material is preheated for 3 to 30 sec before refining, that a refiner pressure is from 4 to 8 bar, preferably 5 to 8 bar and that a refiner rotation speed preferably is above 2000 rpm.

18. A method according to anyone of the preceding claims, characterised in that said fiber material is softwood chips or hardwood chips.

19. A method according to anyone of claims 1-17, characterised in that the fiber material is non-wood fiber material including bagasse, bamboo, reed and straw.

20. A method according to anyone of the preceding claims, characterised in that the pulp obtained after defibration and refining is bleached, preferably with alkaline peroxide, to obtain bleached pulp having high brightness.

21. Mechanical pulp, characterised in that it has been produced according to any one of claims 1-20.

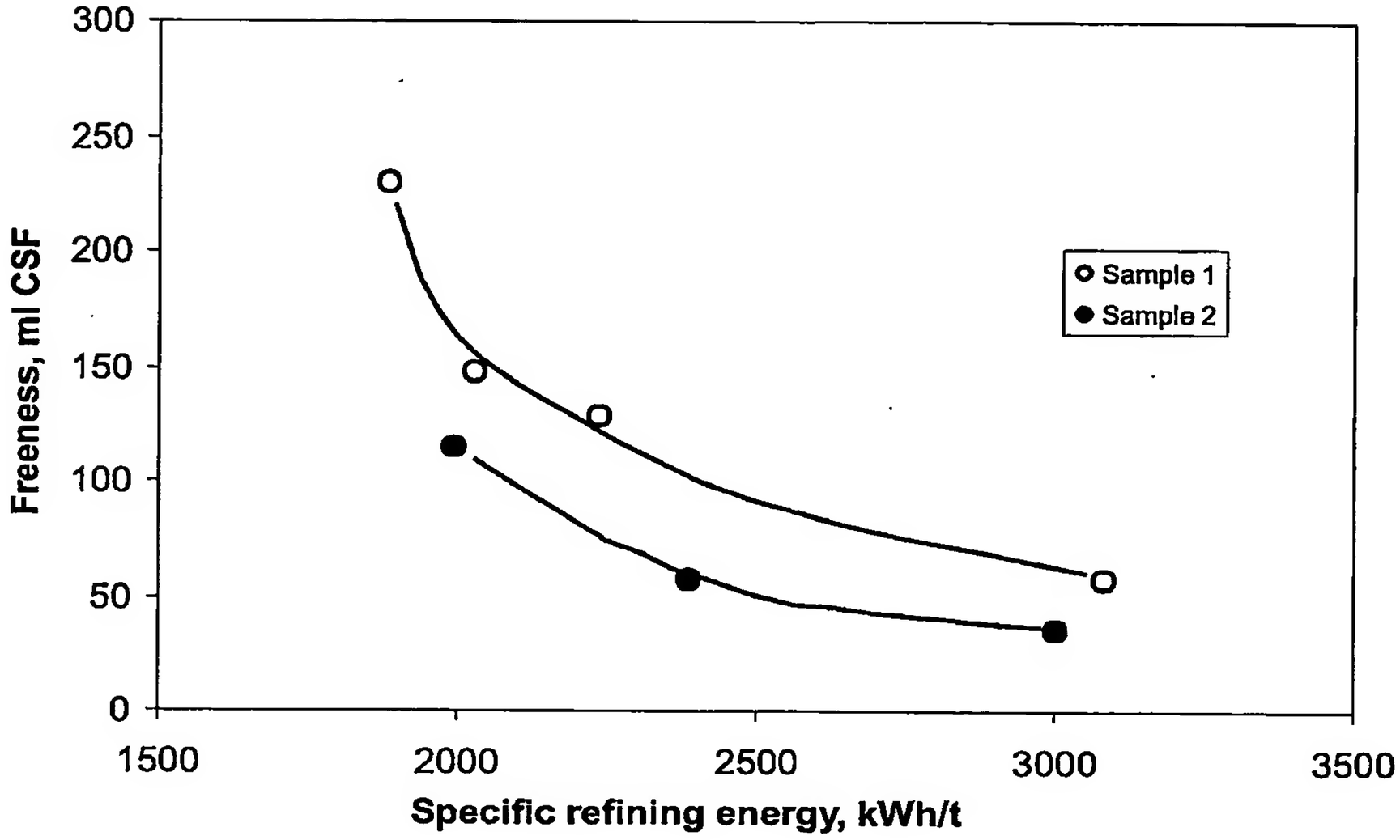

Fig. 1

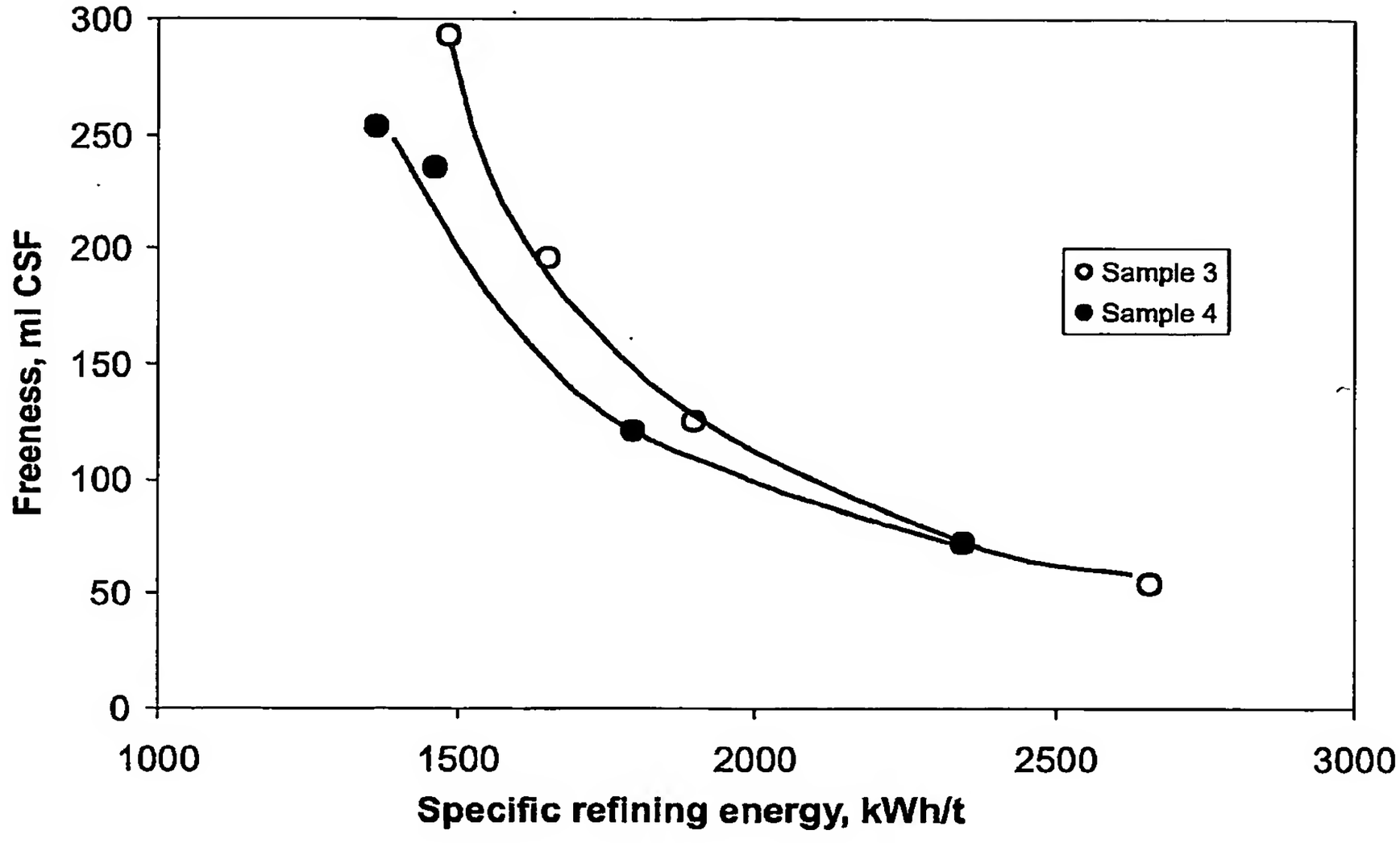

Fig. 2

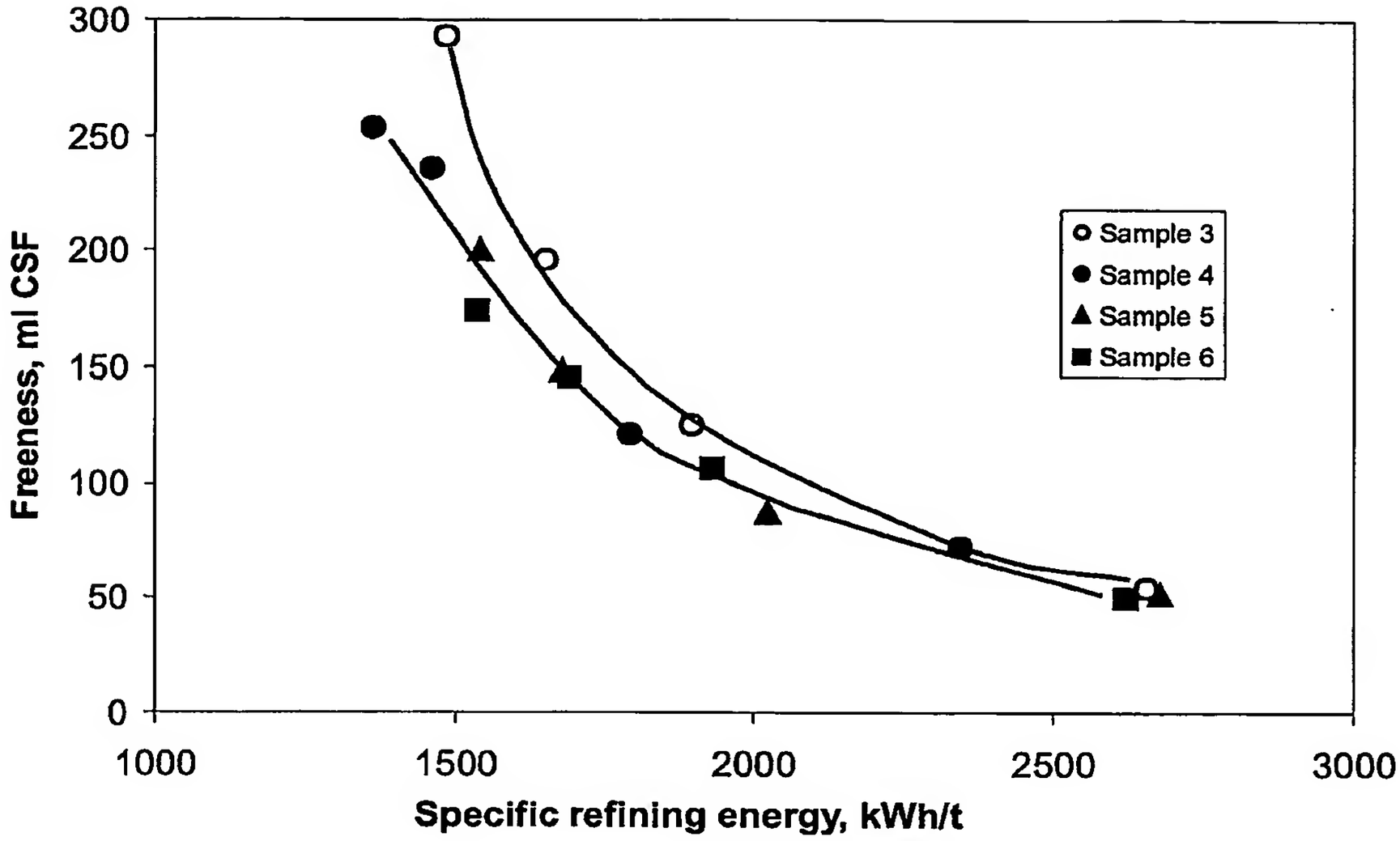

Fig. 3

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE 03/01296

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: D21B 1/02
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: D21B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-INTERNAL, WPI DATA, PAPERCHEM

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0430915 A1 (ENSO-GUTZEIT OY), 5 June 1991 (05.06.91), page 2, line 15 - line 44; page 3, line 6 - line 9, abstract	1,5-9,11-21
Y	--	2-4,10
Y	WO 8902951 A1 (SUNDS DEFIBRATOR AKTIEBOLAG), 6 April 1989 (06.04.89), claims 1-10, abstract	2-4
	--	
Y	WO 9220855 A1 (SUNDS DEFIBRATOR INDUSTRIES AKTIEBOLAG), 26 November 1992 (26.11.92), abstract	10
	--	

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
18 November 2003	24 -11- 2003
Name and mailing address of the ISA/ Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. +46 8 666 02 86	Authorized officer Nina Bergström/MP Telephone No. +46 8 782 25 00

Form PCT/ISA/210 (second sheet) (July 1998)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE 03/01296

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 9806892 A1 (INTERNATIONAL PAPER COMPANY), 19 February 1998 (19.02.98), claim 1, abstract ---	1-21
A	Tappi, Volume 65, No 2, February 1982, Zhung Zhong Lee et al, "Pectic substances contribute to fiber-fiber bonding" page 61 - page 63 ---	1-21
A	US 4891096 A (JEAN-SONY AKKAWI), 2 January 1990 (02.01.90), abstract --- --------	1-21

INTERNATIONAL SEARCH REPORT

Information on patent family members

06/09/03

International application No.
PCT/SE 03/01296

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0430915 A1	05/06/91	CA 2030836 A	28/05/91
		FI 92414 B	29/07/94
		FI 895676 A	28/05/91
		JP 3174079 A	29/07/91
		NO 905134 A	28/05/91
WO 8902951 A1	06/04/89	AU 2525088 A	18/04/89
		SE 461796 B,C	26/03/90
		SE 8703744 A	30/03/89
		SE 8802014 D	00/00/00
WO 9220855 A1	26/11/92	AU 1882492 A	30/12/92
		FI 935205 A	23/11/93
		NO 934229 A	23/11/93
		NZ 242868 A	25/11/94
		SE 468644 B,C	22/02/93
		SE 9101587 A	25/11/92
WO 9806892 A1	19/02/98	AU 4067297 A	06/03/98
		US 6066233 A	23/05/00
US 4891096 A	02/01/90	AT 45398 T	15/08/89
		DE 221811 T	03/09/87
		DE 3664936 D	00/00/00
		DK 158545 B,C	05/06/90
		DK 496186 A	19/04/87
		EP 0221811 A,B	13/05/87
		SE 0221811 T3	
		FR 2588886 A,B	24/04/87
		GR 3000123 T	29/11/90
		GR 88300063 T	18/10/88
		IE 59687 B	23/03/94
		IE 862748 L	18/04/87
		JP 7103484 B	08/11/95
		JP 63501515 T	09/06/88
		PT 83568 A,B	01/11/86
		WO 8702390 A	23/04/87